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(54) Title: HEAT SEALABLE BLEND OF VERY LOW DENSITY POLYETHYLENE OR PLASTOMER WITH POLY-
PROPYLENE BASED POLYMERS AND HEAT SEALABLE FILM AND ARTICLES MADE THEREOF

(57) Abstract

Heat sealable compositions suitable for film and film structures comprise: (a) from 30 to 70 weight percent of a low melting polymer comprising an ethylene based copolymer having a density of from 0.88 g/cm³ to 0.915 g/cm³, a melt index of from 1.5 dg/min to 7.5 dg/min, a molecular weight distribution no greater than 3.5, and a composition distribution breadth index greater than 70 percent; and, (b), being different from (a), from 70 to 30 weight percent of a propylene based polymer having from 88 mole percent to 100 mole percent propylene and from 12 mole percent to 0 mole percent of an alpha-olefin other than propylene.

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are forcefully pressed together at a temperature above the seal initiation temperature of the film. When use is made of equipment such as vertical form, fill and seal machines, the bag is filled with the contents to be packaged while the bottom seal is still hot. Cooling the seal would entail too long a waiting time, thus lengthening the cycle time and increase operating costs. Consequently, the film must be one which enables the formation of a strong seal even as the seal formed is at or near the seal formation temperature.

It is evident that an important characteristic for a heat sealable film is the temperature at which the sealing begins, i.e. the heat seal initiation temperature. It is desired to operate at as low of a temperature as possible because (1) it broadens the heat sealable range, (2) it permits higher productivity due to less time for cooling, (3) it requires less energy to heat seal at lower temperature, and (4) at a lower heat seal initiation temperature, the film is more forgiving of inadequacies in the heat sealing equipment.

Many commonly used plastic materials which are used in the formation of film products could benefit from an improvement of their heat sealing characteristics. For example, crystalline polypropylene films have found extensive use in the field of packaging. Polypropylene films, in both oriented or non-oriented form, are used widely in packaging applications because of their superiority in mechanical properties such as tensile strength, rigidity, surface hardness, and optical properties such as gloss and transparency, and food hygiene such as freedom from toxicity and odor. However, polypropylene films (including those from

polymerization. The metallocene catalysts are also highly flexible in that, by manipulation of catalyst composition and reaction conditions, they can be made to provide polyolefins with controllable molecular weights from as low as about 200 (useful in applications such as lube oil additives) to about 1 million or higher, as for example in ultra high molecular weight linear polyethylene. At the same time, the molecular weight distribution of the polymers can be controlled from extremely narrow (as in a polydispersity, M_w/M_n of about 2), to broad (a polydispersity of about 8).

Exemplary of the development of these metallocene catalysts for the polymerization of ethylene is U.S. Patent No. 4,937,299 to Ewen et al. hereby incorporated by reference. Among other things, this patent teaches that the structure of the metallocene catalyst includes an alumoxane, formed when water reacts with trialkyl aluminum with the release of methane; which alumoxane complexes with the metallocene compound to form the catalyst.

There are a number of structural variables in polyolefins which affect the ultimate properties of the polymer. Two of the most important are composition distribution (CD) and molecular weight distribution (MWD). Composition distribution (CD) refers to the distribution of comonomer between copolymer molecules. This feature relates directly to polymer crystallizability, optical properties, toughness and many other important use characteristics. Molecular weight distribution (MWD) plays a significant role in melt processability as well as the level and balance of physical properties achievable. Molecular weight (MW) determines the level of melt viscosity and the ultimately desired

polymer. In the polymer blend, component (a) is preferably about 30 to about 50 weight percent of the blend based on the total weight of (a) and (b). Suitable copolymers that may be used as blend

5 component (a) include very low density polyethylenes and plastomers which have a density in the range of about 0.88 g/cm³ to about 0.915, g/cm³, a melt index in the range of about 1.5 dg/min to about 7.5 dg/min, a molecular weight distribution (M_w/M_n) of about 1.5

10 to about 3.5, an essentially single melting point in the range of about 60°C to about 115°C, measured as a DSC peak T_m . The term "essentially single melting point" means that at least about 80 wt% of the polymer mass corresponds to that material which is associated

15 to a melting point peak found in the range of 60°C to about 115°C as determined by DSC analysis. The VLDPE or plastomer is a copolymer of ethylene and an olefin other than ethylene and has a composition distribution breadth index about 70% or higher. This parameter

20 distinguishes the VLDPEs and plastomers utilized by this invention from competitive products available commercially. Blend component (b) is preferably about 70 to about 50 weight percent of the blend, based on the total weight of blend component (a) and (b).

25 Blend component (b), which is different from blend component (a), is a propylene based polymer comprising: (i) about 88 mole percent to about 100, e.g., 99 mole percent propylene based on the total moles of olefin and propylene and (ii) about 12 mole

30 percent to about 0, e.g., 1 mole percent of an olefin other than propylene. The polymer blend is formed by mixing blend components (a) and (b) under high shear mixing conditions. A unit such as a twin-screw extruder would be an example of a suitable piece of

Fig. 4 is a DSC curve for a low melting ethylene based copolymer useful as blend component (a).

5 Fig. 5 is a DSC curve for a low melting ethylene based copolymer useful as blend component (a).

Fig. 6 is a DSC curve for a low melting ethylene based copolymer useful as blend component (a).

10 Fig. 7 is a graph of temperature vs. heat seal strength for two neat film samples; the -*---*- line is that of a film prepared from the polymer material of Fig. 3, the -+---+- line is that of a film prepared from the polymer material of Fig. 4.

15 Fig. 8 is a graph of temperature vs. heat seal strength for polypropylene random copolymer/VLDPE blends of various compositions wherein the component (a) of the blend is the polymer of Fig. 4.

20 Fig. 9 is a graph of temperature vs. heat seal strength for neat film of the polypropylene random copolymer utilized as the blend component (b) for the films illustrated in Fig. 8.

25 Fig. 10 is a graph of temperature vs. heat seal strength for polypropylene random copolymer/VLDPE blends of various compositions wherein component (a) is the polymer of Fig. 5.

30 Fig. 11 is a graph of temperature vs. heat seal strength for polypropylene random copolymer/plastomer blends of various compositions where component (a) of the blend is the polymer of Fig. 6.

35 Fig. 12 is a compilation of DSC melting curves of neat polypropylene random copolymer, and blends of the copolymer with varying amounts of a low melting ethylene based copolymer useful as blend

preferably about 50 weight percent propylene based polymer.

The VLDPEs and plastomers utilized as the low melting polymers of the present invention are
5 ethylene/alpha-olefin copolymers wherein the alpha-olefin can have from 3-20 carbon atoms such as ethylene/butene-1, ethylene/hexene-1, ethylene/octene-1, and ethylene/propylene copolymers. These ethylene copolymers with prescribed range of
10 comonomer levels can be prepared by polymerization of the suitable olefins in the presence of supported or unsupported metallocene catalysts systems.

The low melting polymer ingredient utilized in the present invention has a density in the range of
15 about 0.88 g/cm³ to about 0.915 g/cm³. Preferably the density is in the range of about 0.89 g/cm³ to about 0.91 g/cm³. Densities above about 0.90 g/cm³ were measured using standard accepted procedures. At densities below about 0.90 g/cm³, the samples were
20 additionally conditioned by holding them for 48 hours at ambient temperature (23°C), prior to density measurement.

The melt index (MI) of the low melting polymer ingredient of the present invention is in the
25 range of about 1.5 dg/min to about 7.5 dg/min. Preferably the MI is in the range of about 1.7 dg/min to about 5.0 dg/min. MI as measured herein was determined according to ASTM D-1238 (190/2.16). High load MI was determined according to ASTM D-1238
30 (190/21.6).

The low melting polymer ingredient of the present invention has a narrow molecular weight distribution. The ratio of M_w/M_n is generally in the range of about 1.5 to about 3.5. Preferably in the
35 range of about 2.0 to about 3.0.

seal initiation temperature of approximately 120°C and higher before adequate seal strength obtained.

Fig. 1B shows the melting behavior of LDPEs, Plastomers of this invention as the low melting component (Schematic for typical 0.908D VLDPE). The narrow molecular weight distribution (M_w/M_n about 2.0) and narrow composition distribution (CDBI greater than about 70%) ethylene copolymers of this invention offer a melting range close to that desired. Because of the inherent compositional homogeneity of the product, there is predominantly only one melting peak of fairly tight melting range. The peak temperature position can be adjusted by manipulating the crystallinity (or 22650/148-INS density) of the product. Figure 2 illustrates this capability for varying melting peak position. There is a minimum of amorphous, extractable species. These are undesirable in food contact applications.

Fig. 1C shows the desired melting behavior through blending with a lower melting, blend-compatible, component in order to lower seal initiation temperature to a point desirably lower than about 110°C. This figure shows the composite of the blended components. The peak melting point of low melting component should be high enough such that over the temperature range of its melting, a significant amount (i.e., greater than about 15%) of the propylene based polymer is also rendered molten. This fosters good intermolecular mixing of the low melting component with the polypropylene in the melt, leading to stronger seals. The peak melting point of the low melting component should also be low enough so as to allow meaningful reduction of the propylene based polymer's seal initiation temperature, and also, low enough so as to not introduce crystalline species that

should be low enough to allow for applications in the food industry. Generally the percent extractables is in the range of about 2 percent to upwards of 10 percent. Preferably, for food packaging applications, products having extractables 5 percent and under would be utilized.

Hexane extractables is determined according to the U.S. FDA procedure 21 CFR 177.1520(d)(3)(ii), in which a sample is extracted at 50°C in n-hexane (commercial grade) for 2 hours. The filtrate is evaporated and the total residue weighted as a measure of the solvent extractable fraction. A sample size of 2.5 gm 4 mils gauge (0.102 mm), is extracted in 1 liter of solvent.

VLDPEs and plastomers utilized as the low melting polymer of the present invention, comprise ethylene and an alpha-olefin other than ethylene (examples: C₃ to C₈ alpha-olefins). Generally, such a plastomer or VLDPE will comprise in the range of about 85 mole percent to about 96 mole percent ethylene.

The composition distribution breadth index (CDBI) of such VLDPEs and plastomers utilized will generally be in the range of about 70 percent or higher. The CDBI is defined as the weight percent of the copolymer molecules having a comonomer content within 50 percent (i.e., $\pm 50\%$) of the median total molar comonomer content. The CDBI of linear polyethylene, which does not contain a comonomer, is defined to be 100%.

The Composition Distribution Breadth Index (CDBI) is determined via the technique of Temperature Rising Elution Fractionation (TREF). CDBI determination clearly distinguishes, for example, the VLDPE/plastomers of this invention (narrow composition distribution as assessed by CDBI values generally

conditions of polymerization and relatively inert. Preferably, hexane or toluene is employed.

In modification, the polymer components of the present invention may be formed by gas-phase polymerization. A gas-phase process utilizes super-atmospheric pressure and temperatures in the range of about 50° - 120°C. Gas phase polymerization can be performed in a stirred or fluidized bed of catalyst and product particles in a pressure vessel adapted to permit the separation of product particles from unreacted gases. Thermostated ethylene, comonomer, hydrogen and an inert diluent gas such as nitrogen can be introduced or recirculated so as to maintain the particles at a temperature of 50°C - 120°C. Triethylaluminum may be added as needed as a scavenger of water, oxygen, and other adventitious impurities. Polymer product can be withdrawn continuously or semi-continuously at a rate such as to maintain a constant product inventory in the reactor. After polymerization and deactivation of the catalyst, the product polymer can be recovered by any suitable means. In commercial practice, the polymer product can be recovered directly from the gas phase reactor, freed of residual monomer with a nitrogen purge, and used without further deactivation or catalyst removal.

The polyethylene copolymers of the present invention can also be produced in accordance with a high pressure process by polymerizing ethylene in combination with other monomers such as butene-1, hexene-1, octene-1, or 4-methylpentene-1 in the presence of the catalyst system comprising a cyclopentadienyl-transition metal compound and an alumoxane compound. It is important, in the high-pressure process, that the polymerization temperature be above about 120°C but below the decomposition

the range of about 12 mol percent to about 1 mol percent, based on total moles, preferably in the range of about 10 mol percent to about 6 mol percent.

Filmable polypropylene random copolymers useful in the present invention are well known to those of skill in the heat sealing art for polypropylene. An example of a filmable random copolymer polypropylene is Exxon Chemical's Escorene[™] PD-9282. This product is 5 MFR, 5 wt% olefin comonomer. It has a DSC peak melting temperature of about 132°C.

Several commercial polymerization technologies are available to produce these polypropylene random copolymers. These are well known to those of skill in the art of polypropylene polymerization.

The blend composition will generally comprise in the range of about 30 to about 50 weight percent, low melting ingredient and in the range of about 70 to about 50 weight percent of a propylene based polymer both based on the total weight of the low melting ingredient and propylene based polymer. These are preferred ranges. Generally a beneficial contribution from the addition of the low melting ingredient will be noted at a level of about 10 wt%. At a level above 70 wt% low melting ingredient, the blend product shows deficiencies in properties such as rigidity, abrasion resistance and elevated temperature resistance, others.

In some applications, for example in food and flexible packaging, it is important that the amount of haze in the final heat sealable film be minimized so that the film can clearly display the packaged contents and be aesthetically pleasing. In selecting the low melting polymer ingredient and the

The film products made with the blend of the present invention are useful in a wide variety of bag and pouch applications in which heat sealability is important. Bag and pouch forming include, but are not limited to horizontal form-fill-and-seal, and vertical form-fill-and-seal.

Some key properties of the final film are heat sealability and seal strength, hot tack, tensile strength, film rigidity, haze and gloss, extractables, and abrasion resistance.

EXAMPLES

EXAMPLE I

Preparation of VLDPE-1 (1.7 MI, 0.897D, butene-1 Comonomer)

A catalyst is prepared by adding 5.1 liters of a 10% solution of trimethylaluminum in heptane into a dry and oxygen-free two-gallon reactor equipped with a mechanical stirrer. 800 g of undehydrated silica gel, containing 12.3% water, is slowly added into the reactor. After the addition is complete, the mixture is stirred at ambient temperature for one hour. 20 g of di-(n-butylcyclopentadienyl) zirconium dichloride slurried in 30 l of heptane is then added into the reactor and the mixture is allowed to react at ambient temperature for 30 minutes. The reactor is then heated to 65°C, while a nitrogen gas is purged through the reactor to remove the solvent. The nitrogen purging is stopped when the mixture in the reactor turns into a free-flowing powder.

The polymerization was conducted in a 16-inch diameter fluidized gas phase reactor. Ethylene, butene-1 and nitrogen were fed continuously into the reactor to maintain a constant production rate. Product was periodically removed from the reactor to maintain the desired bed weight. The polymerization conditions are shown in the Table below.

VLDPE-1 of this example was prepared according to Example I, and its DSC curve is shown in Fig. 4.

Monolayer film heat seal data of competitive VLDPE B with VLDPE-1 of this invention is shown in Figure 7. Fig. 7 illustrates the seal strength obtained wherein a seal is formed at various heat sealing temperatures in the range of 90°C to 140°C, in 10°C increments. The films were about 1.5 mil gauge produced on a cast film line. Sealing conditions: 14 psi pressure, 1.0 sec. dwell time; Theller Model EB Heat Sealer.

VLDPE-1 provides adequate heat seals (seal strength greater than 1 lb/inch) at a lower temperature than competitive VLDPE B. The final seal strength developed by competitive VLDPE B at the higher temperatures is stronger than that for VLDPE-1; Comonomer type [octene-a vs butene-1] and MW [0.8 MI vs. 1.7 MI] are two reasons, as is well known in the art. Adjustments in these areas can be expected to increase the final seal strength of VLDPE 1 at the higher sealing temperatures.

EXAMPLE III

Melt blends of Exxon's Escorene™ PD-9282 (5 MFR, 95 wt% propylene/5 wt% ethylene) with VLDPE-1 as described in Example I (1.7 MI, 0.897 D, butene-1 comonomer) were prepared on a compounding extruder (1 inch MPM extruder) as follows:

- 80% PD-9282/ 20% VLDPE-1
- 60% PD-9282/ 40% VLDPE-1
- 30% PD-9282/ 70% VLDPE-1

Non-oriented, monolayer cast films of 1.5 mil gauge (Killion lab cast film line) were prepared from each of the 3 blend compositions above, using standard cast film processing conditions. Heat seal measurements performed using sealing conditions described in Example II. Heat seal data shown in Figure 8. Heat seal data on neat PD-9282 (random

Table III

5 Gas phase Polymerization

	Temperature (°F)	139
	Total Pressure (psia)	300
	Gas Velocity (ft/sec)	1.67
	Catalyst Feed Rate (g/hr)	15
10	Production Rate (lb/hr)	20

The polymerized product had a measured Melt Index of 2.1 and a density of 0.906 g/cm³.

The DSC curve for this material is shown in Fig. 5.

15 EXAMPLE V

Melt blends of Exxon's Escorene[®] PD-9282 (5 MFR, 5 wt% comonomer) with VLDPE-2 prepared according to Example IV (2.1 MI, 0.906D, butene-1 comonomer) were prepared on a compounding extruder (1 inch extruder MPM) as follows:

80% PD-9282/20% VLDPE 2

60% PD-9282/40% VLDPE 2

30% PD-9282/70% VLDPE 2

25 Monolayer, non-oriented cast films were prepared from each of the blends and evaluated for heat seal performance, per conditions described previously in Examples II and III. The heat seal data is shown in Figure 10.

30 As observed in Example III, these blend films also provide a favorable balance of sealability at lower temperatures coupled with high seal strength.

EXAMPLE VI

Preparation of Plastomer-1 (3.7 MI 0.8816D, Butene-1 Comonomer)

Table IV

Gas Phase Polymerization

	Temperature (°F)	123
	Total Pressure (psia)	300
5	Gas Velocity (ft/sec)	1.32
	Catalyst Feed Rate (g/hr)	10
	Production Rate (lb/hr)	49

The polymerized product had a weight average molecular weight (M_w) of 81,000 and a polydispersity (M_w/M_n) of 2.4. The density was measured as 0.8816 g/cm³.

The DSC curve for this material is shown in Fig. 6.

EXAMPLE VII

Melt blends of Exxon's Escorene[®] PD-9282 with Plastomer-1 prepared according to Example VI (3.7 MI, 0.8816, butene-1 comonomer) were prepared as follows:

- 80% PD-9282/ 20% Plastomer-1
- 60% PD-9282/ 20% Plastomer-1
- 20 30% PD-9282/ 70% Plastomer-1

As in the previous Examples, heat seal data was generated (Figure 11).

Again, the blends show a favorable balance of sealability at lower temperatures coupled with high seal strength.

EXAMPLE VIII

Film Optical properties, hexane extractables and DSC melting data were measured on the blends described in Examples III, V and VII. Similar data were generated for neat random copolymer polypropylene PD-9282 film, for comparison purposes. This sample is a representation of the product used in the industry today as a heat seal layer for polypropylene films. The data are shown in Table V. DSC melting curves on

TABLE V
NON-ORIENTED CAST FILM PROPERTIES
(BLENDS OF RCP WITH VLDPE-1 AND -2 AND PLASTOMER-2)

	RCP/VLDPE 1 BLENDS	HEXANE EXTRACTABLES (%)	DSC PEAK T _m (°C)	HAZE (%)	GLOSS (%)
5					
	80% PD9282/20%	3.97/3.94	105,131	11	50
10	60% PD9281/40%	3.79/3.91	80,102, 131	19 ⁺	36 ⁺
	30% PD9282/70%	3.27/3.32	78,103, 131	15	43
15	<u>RCP/PLASTOMER-1 BLENDS</u>				
	80% PD9282/20%	3.88/3.90	103,132	16	16
	60% PD9282/40%	9.50/9.23	103,132		
20	30% PD9282/70%	35.9/37.0	63,101, 131		
	<u>RCP/VLDPE-2 BLENDS</u>				
	80% PD9282/20%	3.56/3.59	106,132	11	47
25	60% PD9282/40%	2.79/2.85	90,107, 131	10	49
	30% PD9282/70%	1.85/1.88	89,107, 132	4	73
30	PD9282 5 MFR, 5% C ₂	5.02/5.01	132	13	48
35	40% PD9282/60% XR107L	8.06/8.34	99,132		
	EX300 VLDPE 1.7 MI, 0.897 Density	3.43	77,102		
40	Dowlex VLDPE 0.8 MI, 0.906 Density	4.9	96,123		
45	<hr/> VLDPE-1 is Polymer with 1.7 MI, 0.897 Density				
	<hr/> Plastomer-1 is Polymer with 3.7 MI, 0.8816 Density				
50	<hr/> VLDPE-2 is Polymer with 2.1 MI, 0.906 Density.				
	<hr/> Film thickness on this sample was 2 mil vs 1.6 mil for others during haze/gloss testing.				

Film 2

Core layer: PP-4252 (3 MFR homopolymer)

Skin layers: blend of 80% PD-9282 (random
copolymer polypropylene; 5 MFR,
5 wt% ethylene comonomer) with 20%
competitive VLDPE-B (0.8 MI,
0.906D, octene-1 comonomer)

5
10 The heat seal skin layers of Film 1 contain the narrower molecular weight distribution, narrower composition distribution VLDPE of this invention. The skin layers of Film 2 contain a competitive, more broadly distributed VLDPE. The film data in Table VI show Film 1 to exhibit sealability at lower seal temperatures than Film 2. Also, Film 1 displays
15 better film clarity (lower haze, higher gloss) than Film 2.

This data on RCP/VLDPE blends complements the findings presented in Example II on neat films of VLDPE 1 and competitive VLDPE-B, further illustrating
20 the benefits of narrower MWD, narrower CD VLDPE of this invention in polypropylene heat seal films, versus the more broadly distributed VLDPEs that are common in the industry today.

CLAIMS:

1. A polymer blend composition comprising:
 - (a). from 30 to 70 weight percent of low melting polymer comprising an ethylene based copolymer having a density of from 0.88 g/cm³ to 0.915 g/cm³, a melt index of from 1.5 dg/min to 7.5 dg/min, a molecular weight distribution no greater than 3.5, and a composition distribution breadth index greater than 70 percent; and,
 - (b). from 70 to 30 weight percent of a propylene based polymer having from 88 to 100 mole percent propylene and from 12 to 0 mole percent of an alpha-olefin other than propylene.
2. The composition of claim 1 comprising from 40 to 60 weight percent (a), preferably about 50 weight percent (a).
3. The composition of claim 1 or 2 wherein (a) has an essentially single melting point in the range of from 60 to 115°C.
4. The composition of claim 1, 2, or 3 wherein (a) has a molecular weight distribution no greater than 3, preferably from 2.0 to 3.0.
5. The composition of any preceding claim wherein the comonomer of (a) has from 3-20 carbon atoms, preferably being butene-1, hexene-1, octene-1, or 4-methylpentene-1.
6. The composition of any preceding claim wherein the melt index of (a) is from 1.7 dg/min to 5.0 dg/min.
7. The composition of any preceding claim where (a) or (a) and (b) together has a hexane extractable level of less than 10 percent, preferably less than 5 percent.
8. The composition of any preceding claim wherein at least (a) is prepared using a catalyst